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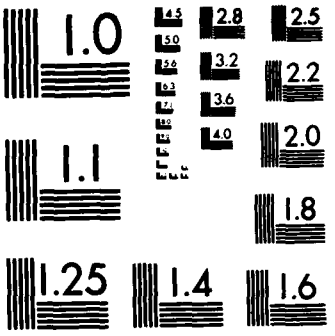

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Annual Technical Report on

Investigation and Synthesis of High Temperature and Increased Stiffness RSP Aluminum Alloys

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The objective of this research is to investigate possible high temperature aluminum alloys useful to 425° C (800° F). A metal matrix composite consisting of an aluminum-magnesium alloy matrix reinforced by spinel (magnesium aluminate) particulate and tri-aluminum (zirconium, vanadium) dispersed in aluminum matrix are being studied. A procedure for preparing specimens of the aluminum alloy matrix-spinel composite using mechanical alloying has been worked out. Extrusions containing 5 volume percent tri-aluminum (0.75 vanadium, 0.25 zirconium) were prepared for this research by Lockheed-Palo Alto from rapidly solidified foil. The measured creep rate at 425° C is much lower than in the current aluminum-iron-cerium alloy.

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SUMMARY

The objective of this research is to investigate two promising systems as the basis for high temperature aluminum alloys useful to 425° C (800° F). The first is a metal matrix composite consisting of an aluminum-magnesium alloy matrix reinforced by spinel (magnesium aluminate) particulate. The second system is tri-aluminum (zirconium, vanadium) dispersed in aluminum matrix. Here the lattice parameter matches that of the matrix. Research on dilute alloys has shown a low coarsening rate for this intermetallic at 425° C. Study of more concentrated alloys with the necessary volume percent of dispersoid for high temperature creep and fatigue resistance is underway.

A procedure for preparing specimens of the aluminum alloy matrix-spinel composite has been worked out and specimens are being prepared. In the procedure rapidly solidified alloy powder and oxide are mechanically alloyed, cold pressed, hot pressed, pressure forged and annealed.

Extrusions containing 5 volume percent tri-aluminum (0.75 vanadium, 0.25 zirconium) were prepared for this research by Lockheed-Palo Alto from rapidly solidified foil. The measured creep rate at 425° C is much lower than in the current aluminum-iron-cerium alloy.

STATEMENT OF WORK

By analogy with Ni-base superalloys which are useful to 0.75 T_m (T_m is the absolute melting temperature of Ni), one may anticipate development of an Al base alloy useful to approximately 425°C or 800°F. In order to develop a successful Al alloy for use at such a temperature, many basic principles must be considered. It is proposed that such an alloy will owe its strength at high as well as room temperature to a uniform dispersion of second phase particles which are coherent or semi-coherent with the matrix. To test this theory, two systems are understudy:

1. Al-Mg alloy matrix - $MgAl_2O_4$ composite
2. Al-Al_3(V,Zr) dispersions

Both of these systems were selected on the basis of lattice matching between an Al alloy matrix and the dispersed phase. Preliminary work was done on Al-Al_3Zr and Al-Al_3(Zr,V) under AFOSR Grant No. 82-0005 where the results on dilute alloys were very promising.

PROGRESS AND RESULTS

1. Al-Mg alloy matrix-$MgAl_2O_4$ spinel composite

The cubic spinel, nominal composition $MgAl_2O_4$, appears to be a better candidate oxide for the dispersion strengthening of Al than the hexagonal oxide α-Al_2O_3. The lattice parameter of stoichiometric $MgAl_2O_4$ is 8.083Å which is almost twice that of Al, 4.0496Å. Thus a semicoherent interface may be anticipated. Furthermore, at least one spinel-structured oxide exhibits some ductility at room temperature.[2] If the dispersed spinel particles in the Al alloy matrix have even very limited ductility, this should result in a much better fracture toughness than with particles like α-Al_2O_3 or SiC which behave in a very brittle fashion. Since the elastic

constants of spinel are considerably larger than those of Al, improved stiffness as well as improved high temperature strength is expected from dispersing spinel in an Al alloy matrix.

To date a procedure for preparing creep and elevated temperature fatigue specimens of Al-Mg alloy matrix-spinel ($MgAl_2O_4$) or corundum (α-Al_2O_3) dispersions has been developed. Al-3% Mg rapidly solidified powder for the matrix was kindly provided for this research by the Alcoa Technical Center. Commercially available spinel and α-Al_2O_3 powders were provided by Baikowski. Composites containing 5 vol.% of either oxide were prepared by mechanical alloying with 1 wt.% oleic acid as a "grinding" aid using alumina pellets as the "grinding" medium. The mechanically alloyed powders were cold pressed to 0.75 in diameter disks with a dual action die at 350 MPa pressure. The cold pressed pellets were heated to 600°C for 5 minutes and then pressure forged in a platen press heated to 300°C to 1 inch diameter. The pressure forged disks were then annealed for 10 hrs at 450°C. The final densities were in the range 99 to 100% of the theoretical. The specimens are cut using electro-discharge machining. Creep and tensile testing of these specimens and microstructural studies are about to begin. Spinel and corundum as dispersoids will be compared.

2. Al-Al_3(V,Zr) dispersions

In previous research completed under AFOSR Grant No. 82-0005C[1] precipitation of the metastable cubic $L1_2$ and tetragonal DO_{23} $Al_3(Zr,V)$ phases from dilute supersaturated solid solutions of Zr and V in Al were investigated. The alloys were prepared and solution treated by arc melting. By this technique it was possible to prepare alloys containing only up to 1 vol.% dispersoid. Addition of V was found to stabilize the cubic $L1_2$ phase (which

is the crystal structure of γ' in the Ni-base superalloys) so that it becomes a candidate dispersed phase for high temperature Al alloys. Both the cubic phase (spherical particles) and the tetragonal phase (platelets) coarsened slowly on isothermal aging at 425°C but the coarsening rate for the cubic phase was a factor of 10 slower in keeping with its lower interfacial energy.

In the present research, 5 vol.% dispersoid alloys obtained from Lockheed, Palo Alto are being investigated. Initially, splats were studied. In the more concentrated alloys cellular precipitation of the cubic phase was more extensive but this mode was suppressed by adding sufficient V. In an alloy designed to give 5 vol.% $Al_3(V_{.875}Zr_{.125})$ only spherical cubic precipitates formed. These coarsened even more slowly than the 1 vol.% alloy which is contrary to the usual effect of increasing the volume fraction on the coarsening rate. Figure 1 compares the volumetric coarsening rate for the 1% $Al_3(V_{.875}Zr_{.125})$ *arc cast* and the 5 vol.% splat. Using the modified Lifshitz-Slyozov-Wagner theory of volume diffusion coarsening due to Brailsford and Wynblatt[3] for 5 vol.% splat cooled samples,

$$\bar{r}_t^3 - \bar{r}_o^3 = \frac{12.8\sigma D V_m^2 C_\infty}{9RT} t = Kt \quad , \qquad (1)$$

an effective interfacial energy 0.13 Joules/m^2 was computed from the measured coarsening rate and the diffusivity of Zr in Al reported in the literature. This is an order of magnitude smaller than computed for the 1% alloy. However, $Al_{10}V$, a stable phase in this alloy, precipitated at grain boundaries during the aging. Its role required further study. Subsequently, Lockheed, Palo Alto prepared rapidly solidified foils and then made bar-shaped extrusions from the foils. Three compositions were prepared, all designed to have 5 vol.% (a) $Al_3(V_{.75}Zr_{.25})$, (b) $Al_3(V_{.5}Zr_{.5})$

and (c) $Al_3(V_{.25}Zr_{.75})$.

Transmission electron microscope study of the 5 vol.% melt spun ribbon with (V/Zr) = (3/1) showed precipitation of mostly spherical cubic Ll_2 particles on aging at 425° C plus a small amount of cellular precipitation of the cubic phase. The coarsening rate of the cubic spherical particles was lower than in the splats, as shown in Fig. 1. The interfacial energy computed from eq.(1) was 0.029 J/m^2, the expected value for a coherent interface between the Ll_2 precipitate and the matrix. It is suggested that defects in the splats lead to accelerated aging. It is also suggested that the coarsening of the 1 vol.% chill cast arc melted alloys was enhanced by dislocations and subgrains. $Al_{10}V$ precipitated at the grain boundaries preventing grain growth although an Ll_2 precipitate free zone formed. Again, long time aging at 425° C (400 hrs) produced a small amount of the tetragonal dispersed phase on dislocations. Decreasing the V/Zr ratio increased the tendency toward cellular precipitation and also increased the amount of precipitation during solidification. This may be related to the fact that V reduces the liquidus temperature of Al-Zr alloys.

The 5 vol.% extruded bars were examined by TEM in the as-extruded condition. Precipitation of the cubic phase occurred during processing. No cellular precipitation was observed in the (V/Zr) = (3/1) alloy as well as no $Al_{10}V$. There was an occasional tetragonal particle. Processing of the lower V/Zr ratio alloys resulted in more cubic cellular and tetragonal precipitation.

In tests by Lockheed, Palo Alto[4] all extruded bars had very satisfactory elongations to fracture at room temperature being in the range 22 to 32%. The tensile strengths were approximately 42 Ksi. A higher tensile strength would, of course, be achieved with a higher volume fraction of dispersed

phase. Exposure of 100 hrs at 425°C had little effect on the elongations to fracture but the ultimate tensile strengths were reduced to 31 Ksi.

Creep tests of the three alloy extrusions were done at 425°C on a dead load creep machine. Creep curves with an initial creep stress P_i= 17 MPa are shown in Fig. 2. For comparison, the creep curve for Al-8.8Fe-3.7Ce alloy, tested by Dr. Lynn Angers, is also given in the figure. The much slower steady state creep rates for alloys 2 and 4 shows that the approach we have adopted as the basis for high temperature alloys is very promising. However, alloy 3 showed poor creep resistance. It entered stage 3 of the creep curve almost immediately after loading indicating a much lower yield stress at 425°C for this alloy. The fractographs show that alloy 3 failed by ductile fracture while alloy 2 and 4 failure by intergranular fracture, the typical fracture mode for creep. TEM examination of foils prepared from crept specimens showed a lower density of Ll_2 phase in alloy 3 (V/Zr = 1) than in alloys 2 (V/Zr = .33) and 4 (V/Zr = 3). Presumably, the Ll_2 phase was transformed to the DO_{23} $Al_3(V_{.5}Zr_{.5})$ and the $Al_{10}V$ phases.

It is an exciting result that alloys 2 and 4 which contain only 5 vol.% of dispersoid show even better creep resistance at 425°C with P_i= 17 MPa than Al-Fe-Ce alloy which contains about 22 vol.% of its dispersoids. It is due to the greater resistance of the Ll_2 $Al_3(Zr,V)$ to the creep deformation at that temperature than the incoherent $Al_{13}Fe_4$ and $Al_{10}Fe_2Ce$ present in the Al-Fe-Ce alloys. Why the Ll_2 $Al_3(Zr,V)$ phase in alloy 3 decomposes faster than that in the alloys 2 or 4 is not known at this time and requires further study.

Future research will be on more concentrated Al base Zr,V alloys being prepared by Lockheed. Richard E. Lewis at Lockheed is a collaborator on the current Al-V-Zr alloy research.

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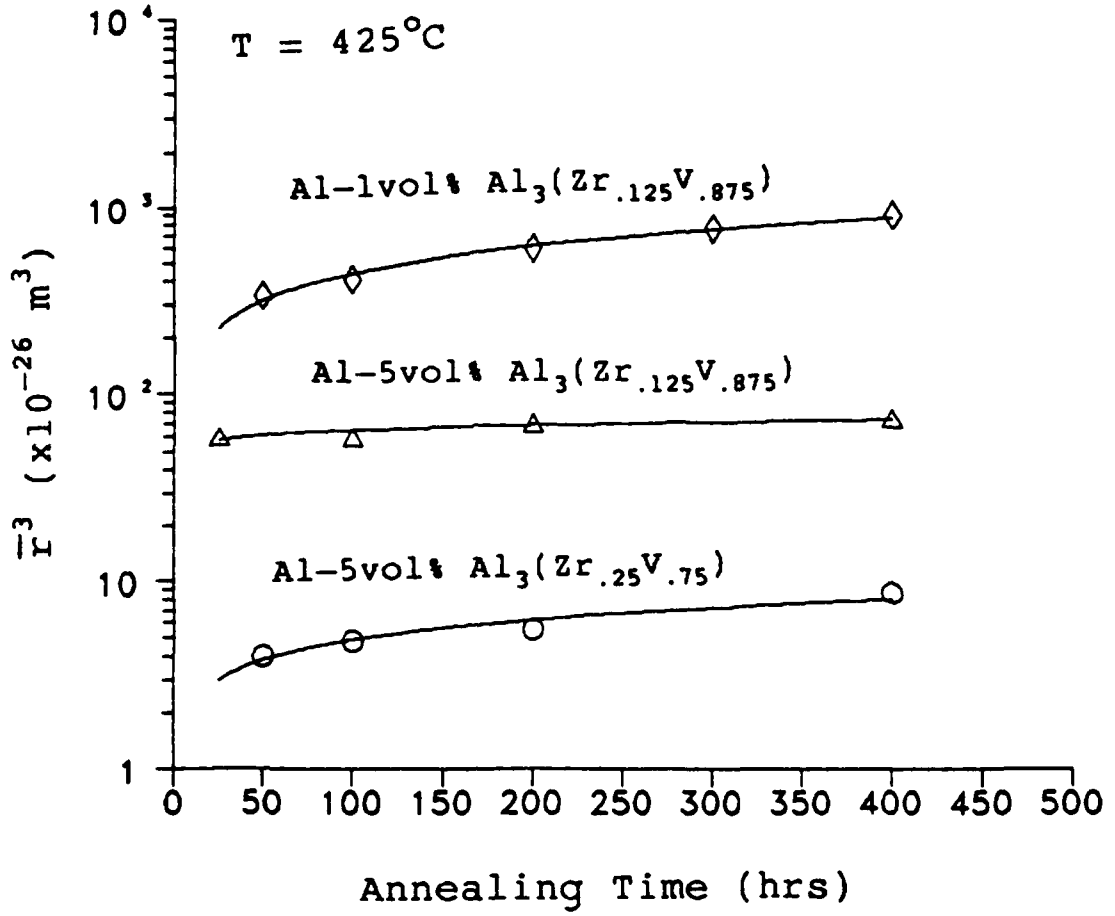
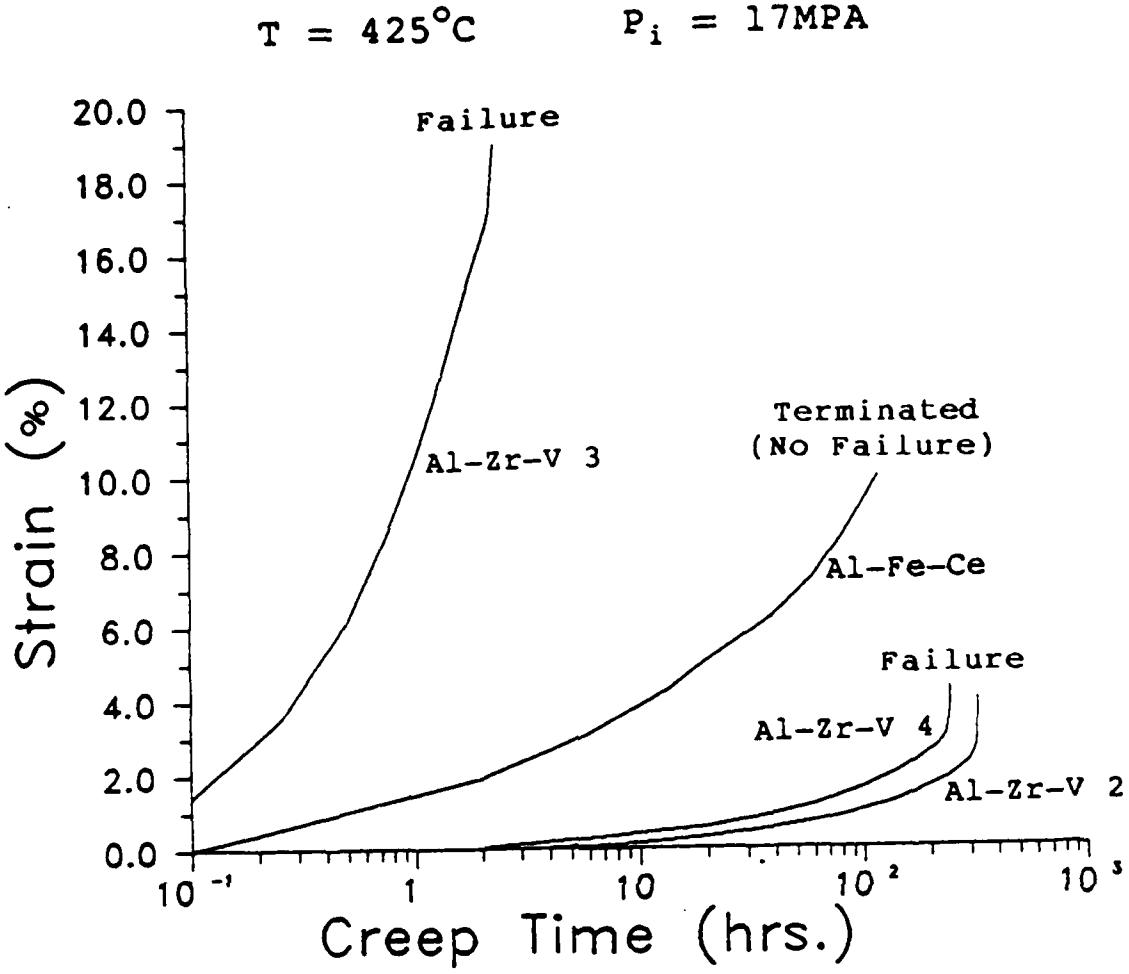


Alloy	Rate Constant K (m^3/hr) – Slope of $\bar{r}^3$ vs t
Al–1vol% $Al_3(Zr_{.125}V_{.875})$	$1.63x10^{-26}$
Al–5vol% $Al_3(Zr_{.125}V_{.875})$	$4.7x10^{-28}$
Al–5vol% $Al_3(Zr_{.25}V_{.75})$	$1.03x10^{-28}$

Fig. 1 Coarsening kinetics of three Al–Zr–V alloys ($\bar{r}^3$ vs t) and their rate constants, K.



Alloy	"Steady state creep rate (1/sec)"
Al-Fe-Ce	1.3×10^{-7}
Al-Zr-V 2	1.7×10^{-8}
Al-Zr-V 3	1.7×10^{-5}
Al-Zr-V 4	3.2×10^{-8}

Fig. 2 Creep curves of Al-Zr-V and Al-Fe-Ce alloys and their steady state creep rates.

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